

FOR USE AT LIBBY OPERABLE UNIT 3 ONLY

**LIBBY OU3 MODIFICATION 1 TO ISO 10312 METHOD
ANALYSIS OF WATER SAMPLES FOR ASBESTOS BY TEM**
Revision 0

Date: May 21, 2009

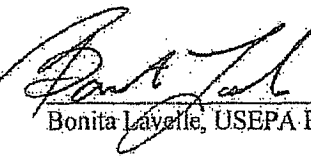
APPROVALS:

TEAM MEMBER

SIGNATURE/TITLE

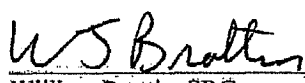
DATE

EPA Remedial Project Manager


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5/22/09

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5/22/09

Revision	Date	Reason for Revision
0	May 21, 2009	--

1.0 PURPOSE

The purpose of this document is to provide modifications to ISO Method 10312 for use at the Libby Superfund Site Operable Unit 3 in the analysis of water samples for Libby Amphibole (LA) by transmission electron microscopy (TEM).

2.0 RESPONSIBILITIES

The Laboratory Director is responsible for ensuring that water samples provided to the laboratory for analysis are prepared and analyzed in accord with the requirements of this modification. It is also the responsibility of the Laboratory Director to communicate the need for any deviations from the modification to the appropriate U.S. Environmental Protection Agency (USEPA) Region 8 Remedial Project Manager or Regional Chemist.

3.0 EQUIPMENT

Sample Preparation

- Sonication device
- Oxygen tank
- Ozone generator
- Plastic and glass tubing

Sample Filtration

- NVLAP-compliant High Efficiency Particulate Air (HEPA) hood
- Particle-free water
- Forceps
- Disposable 47 mm filter funnels
- Side arm filter flask
- Mixed Cellulose Ester (MCE) filters, 47 mm diameter, 0.2 μm and 5.0 μm pore size
- Storage container for filters

Grid preparation and Analysis by TEM

All equipment needed for TEM grid preparation and analysis by TEM analysis is detailed in ISO 10312.

4.0 MODIFICATION SUMMARY

Samples of water from field sampling or laboratory-based studies are transmitted to a qualified laboratory for analysis of asbestos. At the laboratory, aliquots of water are filtered, and the filters are analyzed by TEM in accord with ISO 10312 as specified in the applicable Sampling and Analysis Plan. All results are expressed in units of million fibers per liter (MFL).

5.0 SAMPLE PREPARATION

The project-specific Sampling and Analysis Plans should specify if and how water samples should be prepared for analysis. In some cases, no preparation is needed other than ensuring the sample is well-mixed before filtration. In other cases, it may be appropriate to use sonication to disperse clumps of fibers that may be present, or to use sonication and ozone treatment combined, as detailed in EPA Method 100.1 Step 6.2, especially in samples where microbial growth or other organic matter may be present.

6.0 FILTER PREPARATION

After sample preparation (if needed), one or more aliquots of water from each sample will be filtered through 47 mm MCE filters with 0.2 μm pores, using a backing filter with pore size of 5 μm . The volume of water filtered should be selected to provide a filter loading of about 100-1000 asbestos structures per mm^2 on the filter.

For water samples in which it is possible to estimate the concentration before analysis (e.g., samples from a laboratory-based toxicity test), the appropriate volume may be estimated as follows:

$$\text{Volume (mL)} = \frac{\text{Target Loading (s/mm}^2\text{)} \cdot \text{Effective Filter Area (mm}^2\text{)}}{\text{Expected Concentration (s/mL)}}$$

For example, assuming an effective filter area of 1295 mm^2 , for the analysis of a sample with an expected concentration of 100 MFL ($1\text{E}+05$ s/mL), a loading of about 500 s/ mm^2 would be expected after filtration of about 6 mL.

For water samples for which the concentration can not be reasonably estimated before analysis (e.g., most field samples), then it may be necessary to prepare a series of filters, each with a different volume of water. Typically, this will be done by filtering aliquots of 100 mL, 30 mL, and 10 mL of the sample. Select the filter from the dilution series yielding the largest possible application volume which does not result in an overloaded sample (> 2000 structures per mm^2). If the 10 mL aliquot is overloaded, the laboratory shall prepare a dilution of the sample by removing 5 mL of the remaining volume and diluting to 100 mL. From this secondary dilution, prepare a second series of filters using 60 mL, 20 mL, and 6 mL (corresponding to 3.0 mL, 1.0 mL, and 0.3 mL of the original suspension).

7.0 TEM ANALYSIS

Remove a wedge of about $\frac{1}{4}$ of the sample filter. Prepare at least 4 grids for TEM analysis as detailed in ISO TEM method 10312, also known as ISO 10312:1995(E). Utilize a minimum of 2 grids (typically 3) for analysis, distributing grid openings examined distributed approximately evenly across the grids. Archive the remaining grid(s) and the remaining filter.

Counting rules

All water samples submitted for asbestos analysis by TEM will be analyzed in basic accord with the ISO 10312 counting protocols, with all applicable Libby site-specific Laboratory Modifications, including the most recent versions of modifications LB-000016, LB-000019, LB-000028, LB-000029, LB-000030, LB-000066, and LB-000085.

Stopping Rules

The target analytical sensitivity for sample analysis should be specified in the project-specific SAP. In the absence of such specification, the default target analytical sensitivity for asbestos in water is 50,000 f/L (0.05 MFL). Stopping rules for these analyses are as follows:

1. Calculate the number of grid openings (GOs) needed to achieve the target sensitivity.
2. Count a minimum of 2 GOs in each of 2 grids.
3. Continue counting until one of the following stopping rules is achieved:
 - a. The target sensitivity is achieved
 - b. A total of 50 Libby amphibole (LA) structures have been counted
 - c. A total area of 0.5 mm^2 (usually about 50 GOs) has been examined
4. When one of these rules has been achieved, finish counting the final GO, then stop.

Data Recording and Electronic Data Deliverable

Standard Analysis

Unless otherwise specified in the project-specific SAP, all amphibole structures (including not only LA but all other amphibole asbestos types as well) that have appropriate Selective Area Electron Diffraction (SAED) patterns and Energy Dispersive X-Ray Analysis (EDXA) spectra, and having length $\geq 0.5 \mu\text{m}$ and an aspect ratio (length:width) $\geq 3:1$, will be recorded on the most recent version of the Libby site-specific laboratory bench sheets and EDD spreadsheet ("TEM Water EDD.xls"). Data recording for chrysotile, if observed, is not required.

Rapid Turn-Around Analysis

In some cases, the project-specific SAP may specify that some water samples shall be analyzed using a "rapid turn-around" protocol. The rapid turn-around protocol differs from the standard analysis as follows:

1. Quantitative measurement of length and width is not required for structures that can be readily classified as countable by eye. Measurements may be necessary for structures that are near the size cutoffs for counting (i.e., length close to $0.5 \mu\text{m}$, aspect ratio close to 3:1).
2. Recording of individual structure dimensions and characteristics is not required.
3. Electronic documentation of EDS spectra or SAED patterns is not required.
4. Classification of structures in accord with Libby Laboratory Modification #LB-00066 is not required.

The total number of LA structures observed in each grid opening should be recorded on the most recent version of the Libby site-specific laboratory bench sheets and EDD spreadsheets ("Rapid TEM Water EDD.xls").

8.0 QUALITY CONTROL

The project-specific Sampling and Analysis Plan should specify the types and number of laboratory quality control (QC) samples that should be prepared during the project. In the absence of information in the sampling and Analysis Plan, default guidelines for QC samples are provided in Table 1. This table includes default requirements on the frequency that these QC analyses should be performed, how samples will be selected for QC analyses, the acceptance criteria and corrective actions for these analyses. It is the responsibility of the laboratory manager to ensure that QC requirements are met.

9.0 REFERENCES

International Organization for Standardization (ISO). 1995. Ambient Air – Determination of asbestos fibers – Direct-transfer transmission electron microscopy method. ISO 10312:1995(E).

TABLE 1
LABORATORY QUALITY CONTROL SAMPLE DEFAULT REQUIREMENTS [a]

Lab QC Type & Description	Analysis Frequency [b]	Acceptance Criteria	Corrective Action(s)
Lab Blank A filter that is prepared using laboratory water.	1% (1 per 100 analyses)	No asbestos structures observed in an analysis of 10 GOs.	The laboratory shall immediately investigate the source of the contamination and take steps to eliminate the source of contamination before analysis of any investigative samples may continue.
Repreparation Prepared by applying a second aliquot of sample water to a new filter, which is then prepared and analyzed in the same fashion as the original filter.	2% (1 per 50 analyses) See note [c]	No more than 5% of the original-repreparation pairs are statistically different from each other at the 90% confidence interval. See note [d]	A senior laboratory analyst shall determine the basis of the discordant results, and take appropriate corrective action (e.g., re-training in sample and filter preparation, counting rules, etc).
Recounts Recount Same. A re-examination the same grid openings as were evaluated in the original analysis <u>by the same microscopist</u> who performed the initial examination. Recount Different. A re-examination the same grid openings as were evaluated in the original analysis <u>by a different microscopist within the same laboratory</u> who performed the initial examination.	2% (1 per 50 analyses) See note [c]	See Libby Laboratory Modification LB-000029	A senior laboratory analyst shall determine the basis of the discordant results, and take appropriate corrective action (e.g., re-training in counting rules, etc).
Interlabs A re-examination the same grid openings as were evaluated in the original analysis <u>by a different laboratory</u> who performed the initial examination.	1% (1 per 100 analyses) See note [e]	See Libby Laboratory Modification LB-000029	A senior laboratory analyst at the interlaboratory will contact the originating laboratory to discuss the basis of the discordance. As appropriate, each laboratory will take appropriate corrective action (e.g., re-training in counting rules, etc).

[a] Unless specified otherwise in the project-specific sampling and analysis plan or quality assurance project plan.

[b] When calculating the number of QC analyses required for a project, round up to the nearest whole number.

[c] To be selected by the laboratory in accord with the procedures in Attachment 1 in Libby Laboratory Modification LB-000029.

[d] See Attachment 4 in Libby Laboratory Modification LB-000029 for details on performing this statistical comparison.

[e] To be selected by EPA (or EPA's technical contractor) in accord with the procedures in Attachment 2 in Libby Laboratory Modification LB-000029.

Libby Superfund Site Operable Unit 3 Standard Operating Procedure 3A (Revision 1)

Date: April 27, 2011

OU3 SOP 3A (Rev. 1)

Title: WATER SAMPLING WITH SYRINGE FILTERS

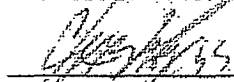
APPROVALS:

TEAM MEMBER

SIGNATURE/TITLE

DATE

EPA Remedial Project Manager



4/27/11

SOP Author

WJ Bratten

4/27/11

REVISION HISTORY

Revision Number	Date	Significant Revisions
0	03/18/2011	--
1	4/27/2011	Add settling step to remove coarse particulates Add detail on tightening of filter holders Changed to 0.2 um pore size filters to decrease time required for filtration

1.0 INTRODUCTION

This Standard Operating procedure (SOP) describes a method for collection and filtration of water samples for the analysis of free (un-bound) Libby Amphibole (LA) asbestos. This SOP is applicable to site water or laboratory water samples collected as part of the Remedial Investigation in Operable Unit 3 (OU3) of the Libby Asbestos Superfund Site.

2.0 HEALTH AND SAFETY WARNING

All personnel engaged in sampling or testing of water containing LA must follow health and safety protocols described in the appropriate health and safety plan. Inhalation exposure to asbestos during field or laboratory work may increase the risk of lung cancer, mesothelioma, asbestosis, and other respiratory diseases.

3.0 RESPONSIBILITIES

This section presents a brief definition of field roles, and the responsibilities generally associated with them. This list is not intended to be comprehensive and often, additional personnel may be involved. Project team member information will be included in project-specific plans (e.g., work plan, field sampling plan, quality assurance plan, etc.), and field personnel will always consult the appropriate documents to determine project-specific roles and responsibilities. In addition, one person may serve in more than one role on any given project.

Project Manager: Specifies the site-specific field or laboratory sampling program, with input from other key project staff and applicable oversight agencies.

Quality Control Manager: Overall management and responsibility for quality assurance and quality control (QA/QC). Specifies QA/QC procedures for the sampling and analytical methods, performs project audits, and ensures that data quality objectives are fulfilled.

Field Team Leader (FTL) and/or Geologist, Hydrogeologist, or Engineer: Implements the sampling program, supervises other sampling personnel, and ensures compliance with SOPs and QA/QC requirements. Prepares daily logs of field or laboratory activities.

Sampling Technician (or other designated personnel): Assists the FTL in the collection and handling of samples. Performs the actual sample collection, packaging, and documentation (e.g., sample label and log sheet, chain-of-custody record, etc).

4.0 SYRINGE FILTRATION PROCEDURE

4.1 Background

Measurement of LA in site or laboratory waters is complicated by the finding that if the water is not completely sterile, organic matter associated with microbial contamination tends to form, which tends to cause the fibers to clump together (EPA 1983a). This causes two effects: a) asbestos fibers that are clumped together are difficult to observe and count using transmission electron microscopy (TEM), and this may lead to a decrease in estimated concentration of LA in the water, and b) fibers within clumps of organic matter tend to adhere to the walls of the sample bottles, thus decreasing the concentration of fibers in the water. The magnitude of these effects is time-variable, and depends on the amount of organic matter present and the time the sample is held before filtering. Both phenomena (fiber clumping, fiber adherence to container walls) have been observed in studies performed to date by EPA at the Libby OU3 site, including a juvenile rainbow trout toxicity test performed using site waters in 2009, and analysis of surface water samples collected at stream sampling station LRC-06 in July 2009.

EPA developed Analytical Method 100.1 (EPA 1983b) for the analysis of total asbestos in water. This method involves treating the water sample with ozone, ultraviolet light, and sonication before filtration. This treatment oxidizes organic material that is present in the water or on the walls of the sample bottle, destroying the material that causes clumping and binding of fibers. Based on studies performed by EPA, this treatment allows good recovery of fibers under a variety of starting conditions.

In some cases, it may also be necessary to measure the concentration of free asbestos in water samples in order to achieve data quality objectives, where “free asbestos” refers to asbestos fibers that exist in the water but are not associated with clumps of organic material.

When seeking to measure the concentration of free asbestos in water, the details of how the water is handled before filtration may be important. For example, if the water sample is placed in a bottle for transport to the laboratory for filtration, it is possible that additional clumping might occur and/or that binding of clumps to the bottle wall might occur before filtration occurs. If so, this could yield results that are not representative of the true concentration of free asbestos in the water at the time of sampling.

One way to avoid this potential problem is to filter the water sample directly at the site of collection, avoiding the lag time needed to transport sample bottles to the laboratory. The purpose of this SOP is to describe a method for the preparation of water filters at the site of collection (field, laboratory) using a syringe filter technique.

4.2 Syringe Filter Sample Collection

4.2.1 Sampling Equipment

The key equipment needed to collect water syringe filter samples is:

- One liter HDPE bottles for collecting water samples in the field
- Plastic syringes, with Luer-Lok fitting, adequate to hold the volume of water specified in the project-specific SAP (typically 10-50 mL)
- Filter holders for 25 mm diameter filter (e.g., Millipore Swinnex SX0002500, or equivalent)
- 25-mm diameter polycarbonate filters with 0.2-um pore size
- 25-mm diameter mixed cellulose ester filters with 5-um pore size
- 25-mm cellulose backing pads
- Tweezers for handling filters
- Filter holder trays for sample storage
- Pre-numbered self-adhesive sample identification labels
- Vinyl electrical tape
- FSDS (see Attachments A and B)

In addition, the following equipment is needed to support the documentation of sampling locations and for sample handling after collection:

- Field notebook, indelible marker
- Global Positioning System (GPS) unit
- Digital Camera
- Chain of custody sheets
- Coolers

4.2.2 Preparing the Filter Holders

Prior to sample collection and filtration, prepare an adequate number of filter holders for use as follows:

1. Unscrew the filter holder
2. Using tweezers, carefully place a 25-mm cellulose backing pad on the filter support in the lower half of the filter holder. On top of this, place a 25-mm MCE filter with 5 um pore size, and on top of that place a 25-mm polycarbonate filter (0.2-um pore size). Ensure that the shiny side of the PC filter is facing upward.
3. Ensure that the Teflon gasket is properly located in the upper portion of the filter holder.
4. Carefully screw the upper portion and the lower portion of the filter holder together. Stop when the halves are firmly finger tight.

4.2.3 Filtration Protocol

Where multiple sampling stations exist along a moving water source (i.e., a creek or drainage channel), water samples generally will be collected from downstream to upstream locations, to minimize the effect of sampling activities on the samples collected.

For collecting syringe filter samples, the procedure outlined below shall be followed.

1. Don appropriate health and safety equipment (if needed)
2. Immediately before sampling, re-tighten each filter holder by firmly grasping the two halves with both hands and twisting firmly.
3. Seal the two filter holder halves together with electrical tape while maintaining tension on the tape.
4. For field samples from OU3, collect a sample of water from the test location in a one liter HDPE bottle. Allow to settle for 2-3 minutes to clear any coarse particulates that may be present. Then, fill a syringe of the appropriate volume (as specified in the project-specific SAP) with water from the upper portion of the bottle.
For water samples collected from laboratory-based toxicity tests, fill the syringe directly from the test chamber. No settling step is required.
5. Turn the syringe upward, and tap to cause any air bubbles to rise to the open end
6. Push the syringe plunger upward, expelling all air bubbles, and adjusting the volume in the syringe to the sample volume specified in the project-specific SAP (typically 10-50 mL).
7. Attach a filter holder containing a 25-mm polycarbonate filter with 0.1-um pore size.
8. Turn the syringe with filter holder downward, and hold as nearly vertical as possible.
9. Using firm pressure, press the syringe plunger downward, forcing all of the water in the syringe through the filter. Do not exert excess pressure, since this may cause water to leak past the filter. The time required to filter a sample is expected to be about 1 minute per 10 mL filtered.
10. Detach the filter holder from the syringe. Maintain the filter orientation. Re-attach a clean 25-mL plastic syringe, with the plunger withdrawn to the 20-mL mark. Holding vertically, press the plunger down, forcing 20 mL of air through the filter and filter holder. Detach the syringe, withdraw the plunger to 20 mL, and force an additional 20 mL of air through the filter and filter holder. This will expel any loose water droplets from the filter holder.
11. Attach a pre-numbered sample identification tag to the filter holder and to the FSDS that records all the relevant data for the sample collected.

4.2.4 Sample Handling

Maintain the filter in the syringe filter holder. Seal the inlet and outlet of the filter holder with vinyl electrical tape to prevent evaporation.

Place the filter holder in a filter holder tray, filter-side up. Place the tray in a cooler, maintaining the filter-side up orientation. Transport or ship the filter holders to the designated analytical laboratory within 24 hours of collection.

4.3 Documentation

At each surface water station, sample details will be recorded on a field sample data sheet (FSDS) form. For field samples, the FSDS provided as Attachment A should be used. For water samples collected as part of a laboratory-based toxicity test, the FSDS provided as Attachment B should be used.

In addition, a field logbook will be maintained in accordance with SOP-9. Data items that shall be recorded in the field logbook include:

- a. Project identification (e.g., OU3 RI Phase 4B)
- b. Location and sample identification, including global positioning system coordinates for field samples (see SOP-11)
- c. Date and time of sample collection
- d. Any deviations from this SOP, and any field conditions that may influence sample quality or relevance

When the sampling activity is completed, the record will be checked by the Project Manager or his/her designee, and the original record will be placed in the project file.

5.0 QUALITY ASSURANCE AND QUALITY CONTROL

Field blank and field duplicate samples will be collected at the frequencies documented in the project-specific sampling and analysis plan.

Field blanks will be prepared by bringing an adequate volume of filtered and deionized (FDI) laboratory water to the sample collection site, and filtering the specified volume (usually 10 mL) through the filter holder.

Field duplicates will be prepared by collecting and filtering a second sample of the same volume from the same location at approximately the same time (within 10 minutes) as the original sample.

6.0 DECONTAMINATION

All syringes and filter holders used in the sampling process shall either be new (un-used) or else shall be thoroughly washed and decontaminated prior to field use and between sample events.

7.0 TRAINING AND PROFICIENCY DEMONSTRATION

All sampling technicians must perform a practice round and demonstrate proficiency prior to collection of authentic field or laboratory syringe filter samples. This will be done as follows:

1. Prepare four syringe filter holders as described in Section 4.2.2
2. Prepare a suspension of India ink by adding about 2.5-10 uL (depending on the strength of the ink) to one liter of water.
3. Pass 20 mL of the Ink dilution through each of the four filters, as described in Section 4.2.3.
4. Remove the filters from the filter holders, and allow to air dry
5. Photograph the filters using a digital camera and transmit the image by e-mail to the analytical laboratory in Libby (mobileasbestoslab@emsl.com) for evaluation.

8.0 REFERENCES

EPA. 1983a. Development of Improved Analytical Techniques for Determination of Asbestos in Water samples. Report prepared for the U.S. Environmental Protection Agency, Environmental Research Laboratory, Office of Research and Development, Athens, GA, by the Ontario Research Foundation, Mississauga, Ontario. EPA-600/4-83-042. September, 1983.

EPA. 1983b. Analytical Method for Determination of Asbestos in Water. U.S. Environmental Protection Agency, Environmental Research Laboratory, Office of Research and Development, Athens GA. EPA-600/4-83-043. September, 1983.

ATTACHMENT A

**FIELD SAMPLING DATA SHEET
FOR SYRINGE FILTER SAMPLES
OF SITE WATERS**

LIBBY OU3 FIELD SAMPLE DATA SHEET

SURFACE WATER AND SEDIMENT

Station ID: _____ Sampling Date: _____
 Field Logbook ID: _____ Logbook Page No: _____
 GPS Coordinate System: UTM Zone 11 North, NAD83 datum, meters
For New Stations Only: X coord: _____ Y coord: _____ Elev: _____
 Sampling Team: MWH Samplers Initials: _____

WATER QUALITY PARAMETERS (if applicable)

Time Measured (hh:mm)	Temp. (°C)	pH	Specific Conductance (mS/cm Auto-comp @ 25°C)	Diss. O ₂ (mg/L)	ORP (mV)	Turbidity (NTU)

SAMPLE COLLECTION

Index ID	AFFIX LABEL HERE	Sampling Time: _____ Sample Type: Field Sample Media : <input type="checkbox"/> Sediment <input type="checkbox"/> SW (aqueous) <input type="checkbox"/> SW (filter) - Vol. Applied _____ mL	Sampling Method (if applicable): Grab or Composite # of Composites: _____ Sampling Depth: Top (in) _____ Bot (in) _____
	AFFIX LABEL HERE	Sampling Time: _____ Sample Type: Field Sample Media : <input type="checkbox"/> Sediment <input type="checkbox"/> SW (aqueous) <input type="checkbox"/> SW (filter) - Vol. Applied _____ mL	Sampling Method (if applicable): Grab or Composite # of Composites: _____ Sampling Depth: Top (in) _____ Bot (in) _____
	AFFIX LABEL HERE	Sampling Time: _____ Sample Type: SP FD MS MSD PE FB TB EB Media : <input type="checkbox"/> Sediment <input type="checkbox"/> SW (aqueous) <input type="checkbox"/> SW (filter) - Vol. Applied _____ mL	Sampling Method (if applicable): Grab or Composite # of Composites: _____ Sampling Depth: Top (in) _____ Bot (in) _____

COMMENTS

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Note: FS Field Sample SP Field Split Sample FD Field Duplicate Sample
 TB Trip Blank Sample MS Matrix Spike Sample MSD Matrix Spike Duplicate Sample
 FB Field Blank Sample EB Equipment Decon Blank Sample PE Performance Evaluation

Sample

Field Data Entered by: _____	Field Entries Checked by: _____
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ATTACHMENT B

**FIELD SAMPLING DATA SHEET
FOR SYRINGE FILTER SAMPLES FROM
LABORATORY-BASED TOXICITY STUDIES**

LIBBY OU3 FIELD SAMPLE DATA SHEET
SYRINGE FILTER SAMPLES FROM TOXICITY STUDIES

Testing laboratory: _____

Study name: _____

Samplers Initials: _____

SAMPLE DATA

	Sample Number	Sample Type	Sample Information
Index ID	AFFIX LABEL HERE	Field sample	Test Chamber: _____ Nominal Concentration: _____
		Field Blank	Sample day: _____ Sample time: _____
		Field duplicate	Sample volume (mL): _____
Index ID	AFFIX LABEL HERE	Field sample	Test Chamber: _____ Nominal Concentration: _____
		Field Blank	Sample day: _____ Sample time: _____
		Field duplicate	Sample volume (mL): _____
Index ID	AFFIX LABEL HERE	Field sample	Test Chamber: _____ Nominal Concentration: _____
		Field Blank	Sample day: _____ Sample time: _____
		Field duplicate	Sample volume (mL): _____
Index ID	AFFIX LABEL HERE	Field sample	Test Chamber: _____ Nominal Concentration: _____
		Field Blank	Sample day: _____ Sample time: _____
		Field duplicate	Sample volume (mL): _____
Index ID	AFFIX LABEL HERE	Field sample	Test Chamber: _____ Nominal Concentration: _____
		Field Blank	Sample day: _____ Sample time: _____
		Field duplicate	Sample volume (mL): _____

COMMENTS

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Data Entered by: _____	Entries Checked by: _____
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Libby Superfund Site Operable Unit 3 Standard Operating Procedure

Date: April 4, 2011

OU3 SOP 8 (Rev. 1)

Title: SAMPLE HANDLING AND SHIPPING

APPROVALS:

TEAM MEMBER

SIGNATURE/TITLE

DATE

EPA Remedial Project Manager

[Signature]

4/5/11

SOP Author

Natalie Row

4/5/11

Revision Number	Date	Reason for Revision
0	09/26/2007	--
1	04/04/2011	Remove shipping peanuts as a viable cushioning material for the shipment of samples

1.0 INTRODUCTION

This standard operating procedure (SOP) is based on MWH SOP-09, Sample Handling and Shipping, Revision 1.0, March 2004, modified for use at the Libby Asbestos Superfund Site OU3. This SOP describes the requirements for sample handling, storage and shipping. The purpose of this SOP is to define sample management activities as performed from the time of sample collection to the time they are received by the laboratory.

2.0 HEALTH AND SAFETY WARNING

All personnel engaged in soil sampling must follow health and safety protocols described in the health and safety plan. Asbestos fibers are thin and long fibers so small that they cannot be seen by the naked eye. Asbestos fibers are easily inhaled when disturbed and when embedded in the lung tissue can cause health problems. Significant exposure to asbestos increases the risk of lung cancer, mesothelioma, asbestosis (non-cancerous lung disease), and other respiratory diseases (ATSDR 2006).

3.0 DEFINITIONS

Chain-of-Custody: An accurate written record of the possession of each sample from the time of collection in the field to the time the sample is received by the designated analytical laboratory.

Sample: Physical evidence collected for environmental measuring and monitoring. For the purposes of this SOP, sample is restricted to solid, aqueous, air, or waste matrices. This SOP does not cover samples collected for lithologic description nor does it include remote sensing imagery or photographs (refer to SOP-9 for field documentation procedures).

Sampler: The individual who collects environmental samples during fieldwork.

4.0 RESPONSIBILITIES

This section presents a brief definition of field roles, and the responsibilities generally associated with them. This list is not intended to be comprehensive and often additional personnel may be involved. Project team member information will be included in project-specific plans (e.g., work plan, field sampling plan (FSP), quality assurance plan, and etc.), and field personnel will always consult the appropriate documents to determine project-specific roles and responsibilities. In addition, one person may serve in more than one role on any given project.

Project Manager: The Project Manager is responsible for ensuring that the requirements for sample management are included in the appropriate project plans. The Project Manager is responsible for coordinating sample management efforts with input from other key project staff and applicable government agencies.

Quality Control Manager: Overall management and responsibility for quality assurance and quality control (QA/QC). Selects QA/QC procedures for the sampling and analytical methods, performs project audits, and ensures that data quality objectives are fulfilled.

Field Team Leader and/or Field Hydrogeologist, Geologist or Engineer: Implements the sampling program, supervises other sampling personnel, and ensures compliance with SOPs and QA/QC requirements. Prepares daily logs of field activities.

Field Technician: Responsible for sample collection, documentation, packaging, and shipping. Assists the FTL and/or geologist, hydrogeologist, or engineer in the implementation of tasks.

5.0 PROCEDURES

5.1 Applicability

The information in this SOP may be used by direct reference or incorporated into project-specific plans. Deviations or modifications to procedures addressed herein must be brought to the attention of, and approved by, applicable government agencies.

5.2 Sample Management

Sample Containers: The sample containers to be used will be dependent on the sample matrix and analyses desired, and are specified in the project FSP. Only certified pre-cleaned sample containers will be used. Sample containers will be filled with adequate headspace (approximately 10 percent) for safe handling upon opening, except containers for volatile organic compound (VOC) analyses, which will be filled completely with no headspace. This no-headspace requirement applies to both soil and groundwater samples.

Once opened, the containers will be used immediately. If the container is used for any reason in the field (e.g., screening) and not sent to the laboratory for analysis, it will be discarded. Prior to discarding the contents of the used container and the container, disposal requirements will be evaluated. When storing before and after sampling, the containers will remain separate from solvents and other volatile organic materials. Sample containers with preservatives added by the laboratory will not be used if held for an extended period on the job site or exposed to extreme heat conditions. Containers will be kept in a cool, dry place. For preserved samples (except VOCs), the pH of the sample will be checked following collection of the sample. If the pH is not at the required level, additional preservative (provided by the laboratory) will be added to the sample container.

Numbering and Labeling: Refer to OU3 SOP-9.

Custody Seals. Custody seals with the date and initials of the sampler will be used on each shipping container to ensure custody. The custody seal will be placed on opposite sides of the cooler across the seam of the lid and the cooler body. Alternatively, if the sample containers are all placed inside a liner bag within the cooler, the custody seal may be placed across the seal of the liner bag inside of the cooler.

Chain-of-Custody: COC procedures require a written record of the possession of individual samples from the time of collection through laboratory analyses. A sample is considered to be in custody if it is:

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- In a person's possession
- In view after being in physical possession
- In a secured condition after having been in physical custody
- In a designated secure area, restricted to authorized personnel

The COC record will be used to document the samples taken and the analyses requested. Refer to SOP-9 Attachment 2 for the OU3-specific COC form. Information recorded by field personnel on the COC record will include the following:

- Sample identifier (Index ID)
- Date and time of collection
- Sample matrix
- Preservation
- Type of analyses requested
- Unique COC number
- Lab being shipped to
- Signature of individuals involved in custody transfer (including date and time of transfer)
- Airbill number (if appropriate)
- Any comments regarding individual samples (e.g., organic vapor meter readings, special instructions).

COC records will be placed in a waterproof plastic bag (e.g., Ziploc®), taped to the inside lid of the cooler or placed at the top of the cooler, and transported with the samples. Signed airbills will serve as evidence of custody transfer between the field sampler and courier, as well as between the courier and laboratory. If a carrier service is used to ship the samples (e.g., Federal Express, etc.), custody will remain with the courier until it is relinquished to the laboratory. Upon receiving the sample cooler, a laboratory representative should sign in the receiving box of the COC, thus establishing custody. The sampler will retain copies of the COC record and airbill.

Sample Preservation/Storage: The requirements for sample preservation are dependent on the desired analyses and the sample matrix, and are specified in the FSP.

5.3 Sample Shipping

The methods and procedures described in this SOP were developed from these sources:

- 49 CFR 173. Shippers – Shippers – General Requirements for Shipping. United States Code of Federal Regulations available online at <http://www.gpoaccess.gov/cfr/index.html>
- 49 CFR 178. Specifications for Packaging. United States Code of Federal Regulations available online at <http://www.gpoaccess.gov/cfr/index.html>
- ASTM D 4220. Standard Practice for Preserving and Transporting Soil Samples. American Society for Testing and Materials available online at <http://www.astm.org/>
- ASTM D 4840. Standard Practice for Sampling Chain-of-Custody Procedures. American Society for Testing and Materials available online at <http://www.astm.org/>

Procedures for packaging and transporting samples to the laboratory are dependent on the chemical, physical, and hazard properties of the material. The procedures may also be based on an estimation of contaminant concentrations/properties in the samples to be shipped. Samples will be identified as environmental samples, excepted quantities samples, limited quantities samples, or standard hazardous materials. Environmental samples are defined as solid or liquid samples collected for chemical or geotechnical analysis. Excepted quantities involve the shipment of a few milliliters of either an acid or base preservative in an otherwise empty sample container. Limited quantities are restricted amounts of hazardous materials that may be shipped in generic, sturdy containers. Standard hazardous material shipments require the use of stamped/certified containers. All samples will be packaged and shipped or hand delivered to the laboratories the same day of sample collection, unless otherwise specified in the project-specific FSPs.

The following paragraphs describe standard shipping procedures for different types of samples. Any exceptions to these procedures will be defined in the FSP. It is the responsibility of the sampler to refer to the U.S. Department of Transportation (DOT) (<http://hazmat.dot.gov/regs/rules.htm>) regulations when dealing with a substance not addressed in this SOP for requirements and limitations associated with the shipment.

Sample Shipping via Commercial Carrier:

Aqueous or Solid Samples: Samples will be packaged and shipped to the laboratories the same day of sample collection, unless otherwise specified in the FSP and depending on holding time requirements for individual samples. For aqueous or solid samples that are shipped to the laboratory via a commercial carrier the following procedures apply:

- Sample labels will be completed and attached to sample containers.
- The samples will be placed upright in a waterproof metal (or equivalent strength plastic) ice chest or cooler.
- For shipments containing samples for volatile organic analysis, include a trip blank.
- Ice in double Ziploc[®] bags (to prevent leakage) will be placed around, among, and on top of the sample bottles. Enough ice will be used so that the samples will be chilled and maintained at $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$ during transport to the laboratory. Dry ice or blue ice will not be used.
- To prevent the sample containers from shifting inside the cooler, the remaining space in the cooler will be filled with inert cushioning material, such as additional bubble pack or cardboard dividers, such that the sample containers remain upright and do not break.
- Tape shut the cooler's drain plug
- The original copy of the completed COC form will be placed in a waterproof plastic bag and taped to the inside of the cooler lid or placed at the top of the cooler.
- The lid will be secured by wrapping strapping tape completely around the cooler in two locations.

- Mark the cooler with arrow labels indicating the proper upright position of the cooler.
- Custody seals consisting of security tape with the date and initials of the sampler will be used on each shipping container to ensure custody. Two signed custody seals will be placed on the cooler, one on the front and one on the back.
- A copy of the COC record and the signed air bill will be retained for the project files.
- Affix a label containing the name and address of the shipper to the outside of the cooler

Hand-Delivered Samples: For aqueous or solid samples that will be hand carried to the laboratory, the same procedures apply.

Excepted Quantities: Usually, corrosive preservatives (e.g., hydrochloric acid, sulfuric acid, nitric acid, or sodium hydroxide) are added to otherwise empty sample bottles by the analytical laboratory prior to shipment to field sites. However, if there is an occasion whereby personnel are required to ship bottles with these undiluted acids or bases, the containers will be shipped in the following manner:

1. Each individual sample container will have not more than 30 milliliters of preservative.
2. Collectively, the preservative in these individual containers will not exceed a volume of 500 milliliters in the same outer box or package.
3. Despite the small quantities, only chemically compatible material may be placed in the same outer box, (e.g., sodium hydroxide, a base, must be packaged separately from the acids).
4. Federal Express will transport nitric acid only in concentrations of 40 percent or less.

5. A "Dangerous Goods in Excepted Quantities" label will be affixed to the outside of the outer box or container. Information required on the label includes:

- Signature of Shipper
- Title of Shipper
- Date
- Name and Address of Shipper
- Check of Applicable Hazard Class
- Listing of UN Numbers for Materials in Hazard Classes

Limited Quantities: Occasionally, it may become necessary to ship known hazardous materials, such as pure or floating product. DOT regulations permit the shipment of many hazardous materials in "sturdy" packages, such as an ice chest or cardboard box (not a specially constructed and certified container), provided the following conditions are met:

1. Each sample bottle is placed in a plastic bag, and the bag is sealed. Each VOC vial will be placed in a sealable bag. As much air as possible is squeezed from the bag before sealing. Bags may be sealed with evidence tape for additional security.
2. Or each bottle is placed in a separate paint can, the paint can is filled with vermiculite, and the lid is affixed to the can. The lid must be sealed with metal clips, filament, or evidence tape. If clips are used, the manufacturer typically recommends six clips.
3. The cans are placed upright in a cooler that has had the drain plug taped shut inside and outside, and the cooler is lined with a large plastic bag. Approximately 1 inch of adsorbent material sufficient to retain any liquid that may be spilled, is placed in the bottom of the liner. Only containers having chemically compatible material may be packaged in each cooler or other outer container.
4. The COC record is sealed inside a plastic bag and placed inside the cooler. The sampler retains one copy of the COC record. The laboratory will be notified if the

sample is suspected of containing any substance for which the laboratory personnel should take safety precautions.

5. The cooler is shut and sealed with strapping tape (filament type) around both ends. Two signed custody seals will be placed on the cooler, one on the front and one on the back. Additional seals may be used if the sampler and/or shipper consider more seals to be necessary. Wide, clear tape will be placed over the seals to ensure against accidental breakage.
6. The following markings are placed on the side of the cooler:
 - Proper Shipping Name (Column B, List of Dangerous Goods, Section 4, IATA Dangerous Goods Regulations [DGR])
 - UN Number (Column A, List of Dangerous Goods, Section 4, IATA DGR)
 - Shipper's name and address
 - Consignee's name and address
 - The words "LIMITED QUANTITY"
 - Hazard Labels (Column E, List of Dangerous Goods, Section 4, IATA DGR)
 - Two Orientation (Arrow) labels placed on opposite sides.
7. The Airbill/Declaration of Dangerous Goods form is completed as follows:
 - Shipper's name and address
 - Consignee's name and address
 - Services, Delivery & Special Handling Instructions
 - Cross out "Cargo Aircraft Only" in the Transport Details Box
 - Cross out "Radioactive" under Shipment Type
 - Nature and Quantity of Dangerous Goods

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- Proper Shipping Name (Column B, List of Dangerous Goods, Section 4, IATA DGR)
- Class or Division (Column C, List of Dangerous Goods, Section 4, IATA DGR)
- UN Number (Column A, List of Dangerous Goods, Section 4, IATA DGR)
- Packing Group (Column F, List of Dangerous Goods, Section 4, IATA DGR)
- Subsidiary Risk, if any (Column D, List of Dangerous Goods, Section 4, IATA DGR)
- Quantity and type of packing (number and type of containers: for example, "3 plastic boxes", and the quantity per container, "2 L", is noted as "3 Plastic boxes X 2 L" This refers to 3 plastic boxes (coolers are referred to as plastic boxes) with 2 liters in each box.
- Packing Instructions (Column G, List of Dangerous Goods, Section 4, IATA DGR).
- Note: Only those Packing Instructions in Column G that begin with the letter "Y" may be used. These refer specifically to the Limited Quantity provisions.
- Authorization (Write in the words Limited Quantity)
- Emergency Telephone Number (List 800-535-5053. This is the number for INFOTRAC.)
- Printed Name and Title, Place and Date, Signature.

Standard Hazardous Materials: Shipment of standard hazardous materials presents the most difficulty and expense. However, there may be occasion whereby a hazardous material cannot be shipped under the Limited Quantity provisions, (e.g., where there is no Packing Instruction in Column G, List of Dangerous Goods, IATA Dangerous Goods Regulations, that is preceded by the letter "Y").

In such cases, the general instructions noted above but for non-Limited Quantity materials will apply, with one important difference: standard hazardous materials shipment requires the use of certified outer shipping containers. These containers have undergone rigid testing and are, therefore, designated by a "UN" stamp on the outside, usually along the bottom of a container's side. The UN stamp is also accompanied by codes specifying container type, packing group rating, gross mass, density, test pressure, year of manufacturer, state of manufacturer, and manufacturer code name. The transport of lithium batteries in Hermit Data Loggers is an example of a standard hazardous material where only a designated outer shipping container may be used.

5.4 Holding Times

The holding times for samples will depend on the analysis and the sample matrix. Refer to the FSP for holding times requirements.

6.0 QUALITY ASSURANCE AND QUALITY CONTROL

All sample shipments must be documented in the field logbooks and/or field forms, including rationales deviations from this SOP. The Field Team Leader or designated QA reviewer will check and verify that handling and shipment documentation has been completed per this procedure and other procedures referenced herein.

7.0 DECONTAMINATION

All shipment coolers shall be maintained clean of sampled material to avoid exposure during shipment. Any investigation-derived waste generated in the sampling process shall be managed in accordance with the procedures outlined in SOP-12.

8.0 REFERENCES

Agency for Toxic Substances and Disease Registry. 2006. Asbestos Exposure and Your Health.

Enforcement Considerations for Evaluations of Uncontrolled Hazardous Waste Disposal Sites by Contractors, Draft, Appendix D, April 1980.

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